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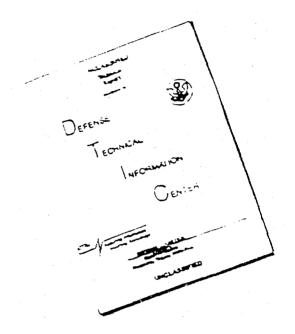
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PARCES TESTS ON POLIVIENT. CALORIDE FIEUR PLASTICIZED MITE TIMEE PLASTICIZEDS

FRANKFORD ARSENAL REPORT NO. 8-900



FUNGUS TESTS ON POLYVINYL CHLORIDE FILMS PLASTICIZED WITH THREE PLASTICIZERS

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Sigmund Berk



SECOND REPORT

PROJECT TMI-1536G

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PLIATE

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PITMAN DUNN LABORATORY Prankford Arbenal Philadelphia, Pa.

January 1049

KEPONT NO R-489

FUNDUS TESTS ON POLYVINYL CHLORIDE FILMS PLASTICIZED WITH

SECOND REPORT

PROJECT TWI-15360

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Previous report

R-697

OBJECT

To determine the susceptibility to fungus growth of polyvinyl chloride films plasticized with dibutyl sebacate; dioctyl phthalate, and butadiene-acrylonitrile, and to determine the effect of the fungus growth on the tensile strength and parcentage elongation of the plastic films.

SUMMARY

Fungus resistance and tensile strength tests were conducted on polyviryl chloride plastic films formulated with three plasticisers: The film with dibutyl sebacate as the plasticiser was very succeptible to Aspergillus flavus and to a species of Penicillium and Trick terms. The dibutyl sebacate plasticised film which was incoulated with the species of Trichederms showed a large increase in the tensile strength and a significant decrease in the parcentage elongation. The increase in tensile strength is attributed to the removal of the placticiser from the film by the mold.

The polyvinyl chloride films formulated with dioctyl phthalate or butadiene-acrylonitrile as the plasticizers showed only slight susceptibility to the species of Trichoderms used. At the end of a 6 weeks incubation period, the two plastic films exhibited only insignificant changes in the tensile strength and percentage elongation.

In so far as resistance to fungi is concerned, it is concluded that dioctyl phthalate and butadiene-acrylonitrile are satisfactory plasticisers for this polyvinyl chloride plastic formulation.

AUTHORIZATION

00 471.873/656, ORDIN: FA 471.8732/5655, 20 Oct 1948

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I. INTRODUCTION

Plastic films were cast from three formulations containing a mixture of polyvinyl chloride resin, plasticizer, polarized oil, light inhibitor, and methyl isobutyl ketons. These dispersions were prepared by a new method involving electronic heating. The polyvinyl chloride dispersion is used hot (80°C-100°C). After casting, the films were aged for two weeks at room temperature to allow the escape of solvents.

The intended use of the polyvinyl chloride dispersion is as a hot dip to close perforations in cartridge cases. Since it is desired that the plastic be fungus resistant, the film plasticized with dibutyl sebacate, the first film available, was tested for susceptibility to mold and possible fungistatic properties (Test 1). Additional polyvinyl chloride plastics plasticized with dioctyl phthalate, or but a diene-acrylonitrite were also rated for their susceptibility to Trichoderms sp. and the effect of such mold growth on the tennile strength and percentage elongation of the films (Tests 2 to 4, inclusive). The plastic formulations containing dibutyl sebacate or dioctyl phthalate had 20% solids. The third formulation containing but addense-acrylonitrile plasticizer had 15% solids.

Dumb-bell specimens 6" long (Fig 20), having a constriction 1/2" wide and 2" long, were cut from the cast films with a metal dia. (1) The dumb-bell specimens were then conditioned at room temperature for an additional two weeks prior to exposure to mold tests.

II. METHODS AND RESULTS

A. Susceptibility to Wold Growth and Fungistatic Properties of a Polyvinyl Chloride Film Plasticized with Dibutyl Sebacate (Test 1).

Forty-ml portions of mineral salts agar(2) with and without l% dextrose were poured into 16-ounce square bottles. The bottles

⁽¹⁾ Federal Specification ZZ-R-601a, 25 June 1940, "Hubber Goods; General Specifications (Nethods of Physical Tests and Chemical Analyses)."

(2) K ₂ HPO ₄	0.7 K.	Fedu _k •7H ₂ O	0.002 д.
KH ₂ PO ₄		ZnSO _k •7H ₂ O	0.002
Mg30), •7H ₂ 0	0.7	MnSO/ Hyō	0.001
NH _L NO ₃	1.0	Agur (Dirco)	15.0
NACL	0.005	Distilled water	1000.0 ml

containing the medium were sterilized at 15 lbs pressure for 20 minutes and placed on their sides. The pH of the autoclaved dextrosefree medium was 6.4. Six-inch tensile specimens of the plastic film plasticised with dibutyl sebacate were placed on the solidified medium in the bottles (Fig 1A).

Three day old cultures of Penicillium sp. U.SD.A 1336.2, Trichoderms sp. FA 69, and Aspergillus flavus SN-3 growing on Difection dextrose agar were used as a source of inoculum. Spore suspensions were prepared by soraping the sporulating surface of the culture with a flamed platinum needle and depositing the spore charge into flasks containing glass beads and 25 ml sterile distilled water. The flasks were agitated to break up spore clumps, and the specimens were inoculated by pipetting 1 ml of the pure suspensions onto the surface of the specimen and the agar medium. Four samples were used with each type of agar and four samples served as uninoculated controls. The bottles containing the samples were incubated for 3 wasks in a constant temperature room maintained at 29.4°C & 0.6°C.

Table I shows that the polyvinyl chloride plastic plasticized with dibutyl sebacate is very susceptible to 3 species of fungi when incubated on a non-mutrient mineral salts agar. In this test, the source of carbon for the growth of the fungi in furnished by the plastic sample. The National Bureau of Standards and the British Ministry of Supply() have reported that pure polyvinyl chloride resists not susceptible to fungal growth. Dibutyl astacate has been reported by the National Bureau of Standards and the Naval Research Laboratory() as moderately susceptible to fungi. The extreme susceptibility to fungus growth of this plastic formulation is attributed to the plasticizer.

The specimens incubated on the mineral salts agar with 1g dextrose produced no inhibition of fungus growth on the nation of on the edges of the specimens. This plastic formulation is therefore considered not fungicidal.

- B. Susceptibility to Trichoderma sp. and the Effect of the mid Growth on the Tensile Strength and Percentage Elemention of Polyvinyl Chloride Films Plasticized with Three Plasticizers.
 - 1. Dibutyl Sebacate (Test 2)

To determine the effect of fungua growth on the tennile at compth of the polyvinyl chloride film planticized with dibutyl

⁽³⁾ Summary Technical Emport of the Tropical Deterioration Administrative Committee, NDEC, "Tropical Deterioration of Equipment and Materials", Vol 1, Chapter 5, 1946

placed on solidified non-nutrient mineral salts agar in square bottles, as described under Test 1. Inoculation of the test strips was effected by pipetting one ml of an aqueous spore suspension of Trichoderma sp. FA69 onto the specimens (Table II, Group A).

An experiment was also conducted to determine the effect of the incubation method, in the absence of fungus growth, on the tensile strength of the plastic strips. A pellet (approximately 0.13 gms) of paraformaldehyde, a volatile fungicide, was placed in a 10 ml beaker and added to each square bottle containing the solidified mineral salts agar (Table II, Group B). The 17 plastic samples comprising this group were not inoculated. Both groups of plastic strips were incubated for 44 days in a constant temperature room maintained at 29.4°C ± 0.6°C.

Table II shows that the strips of polyvinyl chloride plasticised with dibutyl sebacate (Group A) are very susceptible to the species of Trichoderms. Maximum mold growth on the samples incubated without a fungicide crourred after a two week period of incubation. Figure 18 shows that the dibutyl sebacate plasticised film is covered with a dense fungus mat after 3 weeks' incubation.

The fungicide, paraformaldehyde, was not completely effective in inhibiting the growth of conteminating molds present on the plastic strips. Five of the samples had alight to heavy mold growth. More recent work with propyland oxide as a mold-inhibitor for electrical paper tapes incubated in square bottles shows this chemical to be a more effective volatile fungicide.

At . . end of the incubation period both groups of samples were removed from the bottles. The dumb-bell test pieces were dipped in a 0.1% aqueous solution of mercuric chloride for 2-3 minutes, rineed in tap water, and dried at room temperature for 24 hours. After conditioning for 48 hours at 25°C ± 1°C and 50% ± 2% relative humidity, the tensile strength and percentage elongation were determined with a Scott Tensile Tester. Table IX lists the tensile strengths and percentage elongations of the plastic samples. The dumb-bell strips inoculated with the species of Trichoderma (Group A) had a mean tensile strength value of 3462 lbs per square inch, as compared with 2119 for the uninoculated, unincubated controls (Group C), and 1833 for the unincouluted controls incubated with paraformaldehyde (Group B). In terms of percentage change, Group A had increases of 88.6% over Group B controls (incubated with paraformaldahyde) and 66.4% over Group C controls (not incubated). The large increase in the tensile strength of the specimens with mold growth is probably due to the removal of planticizer by the apocies of Trichodorms used. Based on Student's t table, all these gains are significant at the .Ol level. Additional evidence that mold growth produces an increase in tensile strength may be seen in Table II (Group B). The plastic strips that had mold

greent 1st 1 grown of the green to a colthing of the be also green growth in Group 2 also he the highest tensile strength values. The increase in the tensile strength of the plactice insculated with the openies of Trichedorna may be attributed to the distruction or obserption of the plactic for some other equations of the plactic film by the species of mid used.

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The plantic desh-boll speciment insculated with the species of Tricheterus had a mean percentage elementies of 112.75 as compared to 342.76 for the unineutated controls and 20.76 for the unineutated controls insulated with the paraformalishes. In terms of percentage change, droup A had a loss of 57.66 ever droup 3 controls and 67.16 loss over droup 4 controls. The specimene insulated with the paraformalishes had a loss of 18.16 ever droup 6 controls. If only these complex free free maid growth (Table 12, Group 9) are included in the calculations, there is a loss of 18.66 in pircentage elemention. Bood on Mutent's t table, 211 the lesses are significant at the .01 lovel. The lesses in precedure elemention of the plantic film insculated with the species of Trichederum may be attributed to the reserval of planticians by the fungi.

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Table III show the unesoptibility of the polyrings exteride pineticised with disciple pithtiste to mid growth after the 6 week popied of insulation. The extent of mid growth on the films verted from none to slight (Fig 2). The substitution of disciple pithtistes for distiple schools decreased the susceptibility of the polyrings chloride to mid growth.

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growth in Group B also he the highest tensile strength values. The increase in the tensile strength of the plastics inoculated with the species of Trichoderma may be attributed to the destruction or absorption of the plasticiser or some other constituent of the plastic film by the species of mold used.

The plant is samples incubated with the paraformaldehyde had a loss in tensile strength of 13.5% as compared with the uninoculated and unincubated controls. Based on Student's t table, this loss is not significant at .05 level as P=.08. However, if only those samples which did not have mold growth (Table II, Group B) are included in the calculations, the loss in tensile strength is 26.9% over the unincubated controls. This loss is significant at the .01 level and it appears that the paraformaldehyde did have an effect on the tensile strength of the plastic film.

The plantic dumb-bell specimens inoculated with the species of Trichodorms had a mean percentage elongation of 112.9% as compared to 342.9% for the uninoculated controls and 280.9% for the uninoculated controls incubated with the paraformaldehyde. In terms of percentage change, Group A had a loss of 59.8% over Group B controls and 67.1% loss over Group C controls. The specimens incubated with the paraformaldehyde had a loss of 18.1% over Group C controls. If only those samples free from mold growth (Table II, Group B) are included in the calculations, there is a loss of 18.6% in parcentage elongation. Based on Student's t table, all the losses are significant at the .Ol level. The losses in percentage elongation of the plastic film inoculated with the species of Trichodorms may be attributed to the removal of plasticiser by the fungi.

2. Dioctyl Phthalate (Test 3)

Since the polyvinyl chloride plasticised with dibutyl sebacate was found very susceptible to mold growth in Test 2, a formulation containing dioctyl phthalate as the plasticiser was obtained. The National Bureau of Standa.ds(3) found dioctyl phthalate resistant to mold growth. Six-inch dumb-bell tensile specimens of the plastic film were placed on mineral salts agar and inoculated with Trichoderma sp. FA69, as described in Tests 1 and 2.

Table III show the susceptibility of the polyvinyl chloride plasticised with dioctyl phthalate to mold growth after the 6 week period of incubation. The extent of mold growth on the films varied from none to slight (Fig 2). The substitution of dioctyl phthalate for dibutyl sebacate decreased the susceptibility of the polyvinyl chloride to mold growth.

Tensile strength tests were conducted on the conditioned plastic strips, as described in Test 2. Table III lists the tensile

atrength and percentage elemention of the plantic strips uninoculated and inoculated with the species of Trichoderms. The dumb-bell tensile strips inoculated with mold had a mean tensile strength value of 2124 lbs per square inch, as compared with a value of 2264 for the uninoculated and unincubated controls. In terms of percentage change over the unincubated controls, the plantics inoculated with the mold had a 6.2% loss in tensile strength. Based on Student's t table, this loss is not significant at the .01 level but significant at .05 level. There was a 4.8% loss in the mean percentage elongation of the specimens inoculated with mold. This loss is not significant at the .05 level.

3. Butudione-Acrylonitrile (Test 4)

The plantic film planticized with dioctyl phthalate also had properties which were undesirable for its intended use. A formulation was, therefore, obtained which had about 50% polyvinyl chloride and 50% butadiene-acrylonitrile as the planticizer. The solids assumbed to 15%. No information is available on the fungus resistance of this planticizer. Dumb-boll tensile specimens of this plantic were inequalited with the specimen of Trichoderms and incubated on mineral salts agar, as described in Tent 2.

The substitution of but a diene-acrylonitrile for dioctyl phthalate as a plasticizer did not increase the susceptibility of the plastic film to mold growth (Table IV). In Fig 3B, the tensile plastic strip has a trace of growth of the species of Trichoderma. Table IV also lists the tensile strength and percentage elongation of the specimens uninoculated and inoculated with mold. The mean tensile strength of the specimens inoculated with the Trichoderma was 9.9% higher than the unincubated controls. This gain in tensile strength is significant at the .05 level but not significant at the .01 level. There was no significant difference in the mean percentage elongation between the inoculated upocimens and the controls.

III. CONCLUSIONS

- 1. A polyvinyl chloride film plasticized with dibityl sebacate is highly susceptible to sold prowth. The mold growth produces a large increase in the tensile strongth and a large decrease in the percentage eloquation of the plantic film.
- 2. Polyvinyl chloride plantic films plantic files with dioctyl phthalate or initalisms a sylonitrile may be considered mold-inert. The files planticized with those two planticizers had no approximate change in the tenulis strongth and percentage element on after 6 weeks! Incubation with the species of Trichoderms.

TABLE I

Susceptibility to Mold Growth of a Polyvinyl Chloride Plastic Film Containing Dibutyl Sebacate as the Planticizer and Incubated at 29.4° ± 0.6°C for 3 Weeks on Mineral Salt Agar Without and With 1% Dextrose

Inoculuted with	Medium M-Mineral Salte Agar D-Mineral Salte Agar plus 1% Dextrose	Extent of Wold Growth (Average of 4 samples)
Uninoculated Control	M D	1.
Penicillium ap. USDA #1336.2	n D	3
Trichederma sp. FA69	м D	3-4 3-4
Aspergillus flavus SN-3	u D	3

¹ Gods for fungus growths

- 1 = up to 10% of surface moldy
- 2 = 10% to 40% " "
- 3 40% to 75% " "
- 4 = 75% to 100%" " "

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incelates.	sereste serente serente	21.5 28.0 28.0 28.0 28.0 28.0 28.0 28.0 28.0		2719 2809 1664 1331	2201	5 - 601 10 106 at surface - 406 - 756 - 150
Brown B. (With Tarmitares) retyde, S.F. int indialates	4007550 Toliness (8518)	S. S	ភ្នំ ក ធ ដ ភូ ភូ ជ ធ ដ		73.5 8	5 8 2 4 K
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2 "Wicknesses are those of sections seture eroosome

TABLE II a

Soxparizon of kean Tensile Strength and Percentage Elongation of Folywing! Chloride Film Plasticiped withouth Dibutyl Sebscate and Incubaved on Eineral Salts agar

Aroup A compared to Group B compared to Group C Group C Group C Significance & change Significance & change	
Group Ci. Sign	
Group A compared to Group 3 Significance & change	
Group 31	
Smuch 12 (E E E E E E E E E E E E E E E E E E E	والمو
	raile rength

*63.4 Not sig (.05) -13.5 -67.1 Sig (.01) Stg (.a.) Stg (.01.) 21.9.2 342.9 8.65-÷88.5 51g (.al) sig (.a.) Tensile Strength (lim/in²) 3-62-3 1333-6 6.08 112.9 ≤ Klonga-tion

Group 3 was not inoculated but incubated on mineral salts agar for 44 days with paraformaldehyde Group C was not incubated (controls) Group A mas inoculated with Trichoderms up, and incubated on mineral salts agar for 44 days

THE III

and Percentage Elongation of a Folywinyl Chloride File Plasticismod with Dioctyl Fix balate and Incubated at 29.4° ± 0.6°C for 6 meeks on Mineral Salts Agar Susceptibility to Trichoderma sp. F169 and the Effect of the Mold Growth on the Tensile Strength

% Elemention	EXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX	E - 346.2 F - 46.4	x = 361.7 σ = 23.3
Tensile Strength (1bs/in ²)	222 222 222 223 223 223 223 223 233 233	I = 2126.7	X - 2264.4 6- 155.4
Awrage Thickness ² (Mils)	# 4 2 2 2 2 2 3 4 3 2 2 2 2 2 2 2 2 2 2 2		
Kold Growth	+O++O++O+++H=+		ontrola (not incubated)
Sample Musicar	ន្តដ្ឋាន្ត្រី	n = 15	Controls n - 15

1 Code for mald growths

0 = no grout.h

t = trace of growth (less than 3% of surface moldy)
1 = slight growth (3% - 10% of surface moldy)

2 Inichnesses are than of specimens before exposure

TABLE III &

Comparison of bean Tensile Strength and Fercentage Blongation of Polywinyl Chloride Film Flasticised with Dioctyl Phthalate and Incubated on Mineral Salts Agar

Inoculated Compared to Controls Simifficance & change	Sig (.05) -6.2 Not Sig (.05) -4.8
Unimubated Controls	2264.1
Inculated X (rean)	2124.7
	Tensile Strength (lbs/in ²) 2 % Elongation

1 inoculated with Trichodorms sp. and incubated for 6 weeks

Mola growth on the Tensile Strength

Acryloninie	A Mongation	500 525 525 501 501	462.5 462.5 5.2.5	3 3 W W 5 3 K 8 8 8 5	5 8 E	x = 4(3-3	
Effect of the Wold Inducto of the formation ide Plastic Film Containing Estadiene Acryloning Office Films Containing Estadiene Acryloning of for 6 Weeks on Mineral Salts Agar	Tensile Strength (10s/in2)	2181 1545 2000 2052	87 55 7 7 55 7 7 55 7 7 55 7	1521 5705 1771 1833	\$62 \$62 \$62 \$62 \$62 \$62 \$62 \$62 \$62 \$62	x = 2022.9	X = 1840.6
sp. FA69 and the splaying Chlored at 29.4° ± 0.6°	Average Thickness (Mils)	ជជនរ	: শ্রন	- ទ៨¤៨៨	ឧឧଶ		
Lage Slongation of a	()	O 44 40	א ני ני	ପ ୬ ୩ ୦ ୦	5 4 4 C		rols (not incubated)
	3		ጓ 焦Α∂	ነኮይጸቋ/	7745		2attro2

⁻ Code for mold growth:

^{0 =} no growth t = trace of growth (less than % of surface moldy)

² Thicknesses are those of specimens before exposure

TABLE IV a

lemparison of Kean Tensile Strength and Fercentage Elongation of Polyvinyl Chloride Fila Flasticized with Establent-Acrylonitrile and Incubated on Mineral Salts Agar

Significance & change	Sig (.05) +9.9 Not Sig (.05) +0.7
Unincubated Confirols	14.0.6 0.07.1
Incculated X Bean	2022.9
	ensile Strength (lbs/in ²) ; Elongation

I Inoculated with Trichoderms sp. and incubated for 6 weeks





CHONANCE LABORATORY FRANKFORD ARSENCE 11/23/48 RE JIESE

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PENTY SAPITY OF TRICOCERNA SP., OR TENSILE S'ECINEN INCHRATED ON NINERAL SALTS AGAR IN STUARE BATTLE FOR 9 WEEKS POLIVINAL CALGRIDE PLASTIC FILM PLASTICIZED BITE BEBRIYL SEBACATE

WIEW OF SPECIMEN AFTER SEMONAL PRIN BOTTLE

. BET BORNTER SPECTIBER

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CAD DEPT

ONDMANCE LABOR STORY, FRANKFORD ARSENAL.

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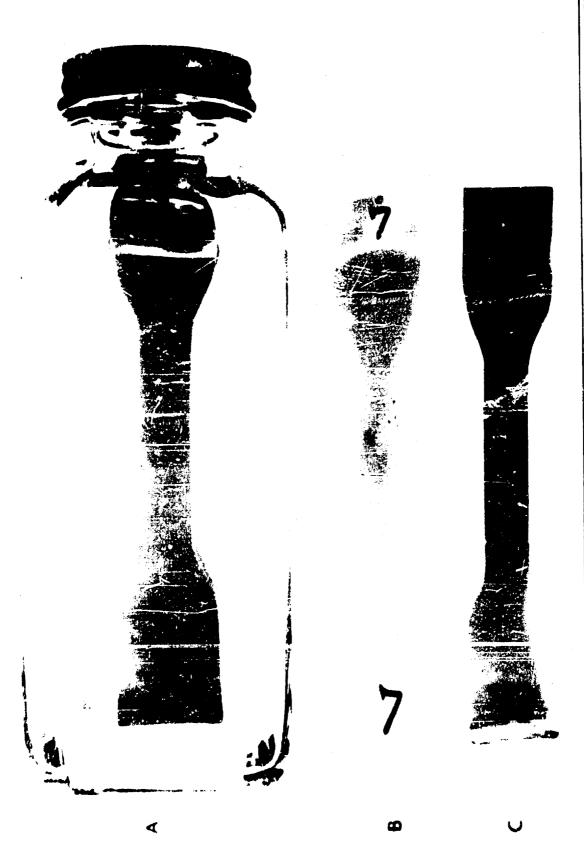
FIGURE 2
PRINTER CHARGE FLASTIC FILE PLASTICIZES SITE SIBCTVL PSTEALATE

A. SPECIMEN INSCRIATES WITH TRICHORERM SP., AND INCHRATED OR MINERAL SALTS AGAR IN SPEARE DOTTLES FUR 9 WEEKS

SAMPLE MEMPRES FING PUTTLE SHOUTHS A TRACE OF MOLS GROWTH

C. BETHCHBATES SPECIMES





11/29/48 OPDMANCE LABORATORY, FRANKFORD ARSENAL

A. SPECIAEW IMPORTATED BITH TYTOPOGENNA SF. AND INCOMATED DE MINERAL SAUTS AGAR IN STRANE MOTHLES FOR 9 WEEKS POLYVING CHASTIS PLASTIC FILB PLASTICIZES SITS BETABLENE ACKTUMITALE

SAMPLE REMOVES FROM SOTTLE SHORTING A TRACE OF HOLD GROWTH

MINCHATES SPECIES